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PATENT KANTOOR
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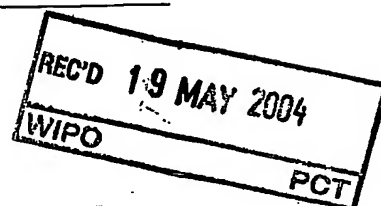


PCT/IB04/01102
Certificate

REPUBLIC OF SOUTH AFRICA

PATENT OFFICE
DEPARTMENT OF TRADE AND
INDUSTRY

Hiermee word gesertifiseer dat
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the documents attached hereto are true copies of Forms P1, P2
and provisional specification and drawing of South African Patent Application
No: 2003/5682 in the name of Lonmin PLC

Filed : 23 July 2003
Entitled : Recovery of Platinum
Group Metals

PRIORITY DOCUMENT

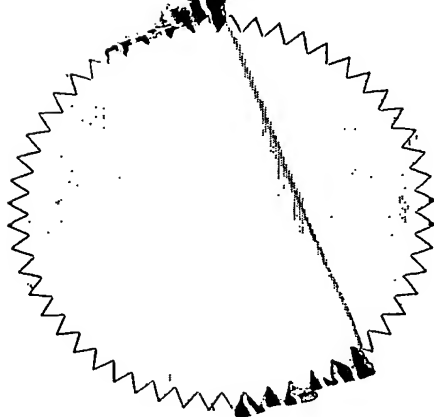
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in die Republiek van Suid-Afrika, hierdie
in the Republic of South Africa, this

4th dag van
May 2004
day of

Registrar of Patents



REPUBLIC OF SOUTH AFRICA			REGISTER OF PATENTS			PATENTS ACT, 1978		
OFFICIAL APPLICATION			LODGING DATE: PROVISIONAL			ACCEPTANCE DATE		
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INTERNATIONAL CLASSIFICATION			LODGING DATE: COMPLETE			GRANTED DATE		
51			23					
FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)								
71	LONMIN PLC							
APPLICANTS SUBSTITUTED:						DATE REGISTERED		
71.								
ASSIGNEE(S)								
71							DATE REGISTERED	
FULL NAME(S) OF INVENTOR(S)								
72	DUNN, GRENVIL MARQUIS BAX, ALAN LEWINS, JOHN DEREK							
CORRECTIONS								
PRIORITY CLAIMED		COUNTRY		NUMBER		DATE		
N.B. Use International abbreviation for country (see Schedule 4)		33	NIL	31	NIL	32	NIL	
TITLE OF INVENTION								
54	RECOVERY OF PLATINUM GROUP METALS							
ADDRESS OF APPLICANT(S)/PATENTEE(S)								
4 GROSVENOR PLACE, LONDON, SW1X 7YL, UNITED KINGDOM								
ADDRESS FOR SERVICE						S & F REF		
74	SPOOR & FISHER, SANDTON					PA135601/P		
PATENT OF ADDITION NO.				DATE OF ANY CHANGE				
61								
FRESH APPLICATION BASED ON				DATE OF ANY CHANGE				

SPOOR & FISHER

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT
AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) - Regulation 22)

23.07.03

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PROSTATE

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

REPUBLIC VAN SUID AFRIKA
S & F REFERENCE

OFFICIAL APPLICATION NO.

21 01. 2003/5682

PA135601/P

FULL NAME(S) OF APPLICANT(S)

71 LONMIN PLC

ADDRESS(ES) OF APPLICANT(S)

4 GROSVENOR PLACE, LONDON, SW1X 7YL, UNITED KINGDOM

TITLE OF INVENTION

54 RECOVERY OF PLATINUM GROUP METALS

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

COUNTRY: NIL NUMBER: NIL DATE: NIL

THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

21 01

THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

21* 01

THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional specification of 22 pages.
- ☒ 2. Drawings of 6 sheets.
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. Assignment of invention.
- ☐ 6. Certified priority document.
- ☐ 7. Translation of the priority document.
- ☐ 8. Assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No.
- ☐ 10. Declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.
- ☐ 14. Other.

74 ADDRESS FOR SERVICE: SPOOR & FISHER, SANDTON

Dated: 23 July 2003

SPOOR & FISHER
PATENT ATTORNEYS FOR THE APPLICANT(S)

RECEIVED
REGISTRAR OF PATENTS DESIGNS, TRADE MARKS AND COPYRIGHT
2003 -07- 23
REGISTRAR OF PATENTS REGISTRATEUR VAN PATENTE, MODELLE, HANDELSMERKE EN OUTEURSREG

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978**PROVISIONAL SPECIFICATION**

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

21	01	2003/5682
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LODGING DATE

22	23 JULY 2003
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FULL NAMES OF APPLICANTS

71	LONMIN PLC
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FULL NAMES OF INVENTORS

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CORRECTIONS

TITLE OF INVENTION

54	RECOVERY OF PLATINUM GROUP METALS
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BACKGROUND OF THE INVENTION

This Invention relates to a method of recovery of platinum group metals (PGMs) from a solution or leachate containing the PGMs and base metals, and to the production of a PGM concentrate suitable as a feed stock to a PGM refinery.

PGMs usually occur as discrete minerals, as dilute solid solutions in sulphide minerals, or are associated with silicates and/or chromitites. The processing of these materials consists of a combination of several unit operations, which ultimately produce individual PGMs – platinum, palladium, rhodium, ruthenium, iridium and osmium - plus gold and silver.

Currently PGM ores containing varying amounts of base metals, such as copper, nickel and cobalt, are crushed, milled and floated in flotation cells using reagents typically used for the recovery of base metal sulphides, to produce a low grade PGM concentrate. This flotation concentrate is dried and then smelted in an electric arc furnace where the PGMs are collected in the matte phase.

The molten furnace matte is transferred to a converter where flux is added and air blown into the bath so as to remove the iron as a slag. During the blowing process, the majority of the sulphur in the furnace matte is converted to sulphur dioxide and emitted as a gas.

The converter matte produced from the converting stage is either granulated or slow cooled so as to form a PGM containing magnetic phase. This matte is then treated in a base metal refinery where it is acid leached or magnetically separated followed by acid leaching. Up to and including the smelting process the PGMs are associated with the base metals during the refining process the PGMs are separated from the base metals to produce a PGM concentrate and the base metals are removed as by-products such as nickel cathodes, nickel sulphate crystals, copper cathodes and cobalt sulphate.

The high grade PGM concentrate is then treated in a precious metal refinery where the individual PGMs are separated and produced in pure metallic form.

A simplified flow sheet of the above process is shown in Figure 1. This conventional processing route for PGMs has its limitations. It is considered suitable for sulphide containing ores from which relatively low quantities of flotation concentrates are produced with high recoveries of PGMs that can be treated economically through a smelter. However, ores that have been oxidised produce greater amounts of flotation concentrates to achieve the same PGM recoveries and this can lead to uneconomic smelting operations.

Concentrates containing relatively high levels of chromite also adversely affect smelting operations, resulting in high costs and lower recoveries.

The erection of new smelters to process PGM containing flotation concentrates has been significantly curtailed in recent years and has all but now been stopped due to very strict environmental legislation and prohibitively high capital costs. This means that any new operation will have to ship flotation concentrates excessively large distances to existing smelters, at high cost, leading to increased metal pipelines.

Environmental legislation is also forcing existing smelters to continually reduce the amount of sulphur dioxide being emitted to the atmosphere. This leads to on sulphur dioxide (SO_2) fixation plants and often with no revenue return. Significant benefit may therefore accrue were the smelting step to be eliminated.

SUMMARY OF THE INVENTION

A process of recovering platinum group metals (PGMs) from a solution or leachate containing PGM values and base metals, includes the steps of:

- a) non-selective precipitation of the PGM values and base metals to form an insoluble precipitate comprising the PGM values and base metals;

- b) selective leaching of the precipitate to form (i) a leach solution containing the base metals and a residue containing the PGM values, or (ii) a leach solution containing the base metals and PGM values and a depleted residue; and
- c) recovery of the base metals from the leach solution of b)(i) or b)(ii) and recovery of the PGM values from the residue of b)(i) or the leach solution of b)(ii), depending on the selective leaching in step b).

The solution or leachate containing the PGM values and base metals is preferably a cyanide solution or leachate.

The non-selective precipitation of the PGM values and base metals in step a) is preferably carried out by controlled acidification of the solution or leachate, in particular by controlled addition of sulphuric acid at a constant low pH.

In one embodiment of the invention, the selective leaching in step b) is a pressure leaching step in which the PGM values remain in the residue. The PGM values are typically recovered from the residue by a fusion step or a further leach step to produce a concentrate rich in PGM values. The base metals are recovered from the leach solution, preferably through precipitation.

In an alternative embodiment of the invention, the insoluble precipitate of step a) is first calcined and then selectively leached to remove the base metals, producing a PGM rich residue or concentrate. Again the base metals are preferably recovered from the leach solution by precipitation.

In a further alternative embodiment of the invention, the insoluble precipitate of step a) is fumed with acid, followed by selective leaching to form a leach solution containing both the PGM values as anions and the base metals as cations, and recovery of the PGM values on an ion exchange resin, which is preferably incinerated to form an ash rich in PGM values or eluted to recover the PGM values. The base metals are preferably recovered from the leach solution by precipitation.

Any PGM values remaining in solution after the non-selective precipitation in step a) may be recovered by passing the solution through an ion exchange resin, as discussed above. Any base metals remaining in the solution may also be recovered, preferably by precipitation.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings, in which:

- Figure 1** is a flow sheet showing a prior art method of recovering PGMs;
- Figure 2** is a flow sheet of an embodiment of the process of the present invention;
- Figure 3** is a flow sheet of alternative embodiments of the process of the present invention;
- Figure 4** is a graph that shows the effect of using NaHS over a range of pHs to precipitate PGMs and base metals;
- Figure 5** is a graph that shows the effect of using H_2O_2 over a range of pHs on recoveries of PGMs and base metals;
- Figure 6** is a graph that shows the effect of a combination of H_2O_2 and $Na_2S_2O_5$ at varying pHs on recoveries of PGMs and base metals;
- Figure 7** is a graph that shows the effect of using sulphuric acid over a range of pHs on recoveries of PGMs and base metals from cyanide leachate; and
- Figure 8** is a graph that shows the effect of temperature of calcination on the leaching extraction of PGMs and base metals.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The present invention provides a process to recover PGMs and base metals from a solution or leachate containing them, in particular a cyanide solution or leachate.

The cyanide leachate is produced through a known cyanide leach process. In this process, a flotation concentrate or an acid pressure leach residue material bearing platinum group metals such as platinum, palladium, rhodium, ruthenium, iridium and osmium, other precious metals such as gold and silver, and various base metals such as copper, nickel and cobalt, is contacted with a cyanide leach solution to solubilise the PGMs and the base metals. The resultant cyanide leachate may, if desired, be concentrated to form a concentrate rich in PGM and base metal complexes.

Referring to Figure 2, in a first stage of the preferred process of the invention, the cyanide leachate is acidified with diluted sulphuric acid in a pH range of 0 – 5,0 and conducted at a temperature in the range of ambient to 80°C. The acidification is preferably done using 1:1 sulphuric acid at around pH 2 and 45°C. The acidification step can take up to 6 hrs.

The precipitation of the PGMs and base metals during acidification may be aided by controlled rates of addition of reagents such as sulphides in the form of sodium sulphide and sodium hydrogen sulphide or oxidants such as hydrogen peroxide and salts of peroxomonosulphuric acid (Caros acid).

After acidification, the precipitated metals are recovered from solution by solid-liquid separation. Any cyanide released as hydrogen cyanide can be recovered in a scrubber.

The solution from the solid-liquid separation is passed through ion exchange columns where it is contacted with a resin that has functional groups that have stronger affinity for PGM complexes than the base metal complexes e.g. certain ammine groups. The PGM complexes bind to the resin in preference to certain base metal complexes.

The ion exchange columns are typically arranged in a "lead-trail" configuration. As the lead column becomes exhausted by the loading of the PGM and base metal complexes, the PGM complexes displace some of the base metals as additional feed is provided to the lead column. The trail column(s) captures these base metal complexes, as the case may be, and so a partition is achieved between the PGMs and the base metals.

To recover the PGM values from the lead PGM column, a number of options are possible. The resin may be removed and ashed to produce a PGM product. Alternatively the resin may be eluted using an appropriate eluant to remove the PGM values.

To assist with the recovery of gold (Au), an ion exchange column loaded with a resin such as activated carbon is typically installed in the system.

Once the solution has passed through the ion exchange columns, the residual base metal values are preferably precipitated from solution using reagents such as caustic soda and/or sodium carbonate and sodium sulphide. The precipitated base metal products can then be recovered from solution by solid-liquid separation. Alternative methods such as ion exchange, solvent extraction and electrowinning can also be used to recover the base metal values.

The filtered precipitated residue from the initial step of acidification, also referred to as the acidification residue, may be repulped using filtrate from a later filtration step. Sulphuric acid is typically added to the repulping step. Any off-gas is scrubbed to recover hydrogen cyanide (HCN), which is recycled to the cyanide leaching stage.

The slurry from the repulping step is fed to a high-pressure vessel, typically a multi-compartment autoclave, where it is heated to a temperature of 140-220°C, preferably between 175 and 185°C. Oxygen is added counter currently to flush off any HCN into the vent from where it may be scrubbed. The autoclave is steam heated, directly or indirectly, and the pressure in the autoclave is maintained typically at 1500 kpa(g) and a retention time of up to 3 hrs. The pulp density can be such as to yield a base metal concentrate varying from 10 to 180 g/l combined base metals, preferably 40 – 80 g/l.

The discharge from the autoclave is cooled to approx 85°C and flocculants such as Flocculant M351 added to aid settling before solid-liquid separation such as thickening and pressure filtration.

The filtrate from the solid-liquid separation step can be treated by a variety of means to recover the base metals. These include:

- Copper solvent extraction to remove copper.
- Subject to the copper nickel ratio in the leachate, metallurgically remove the copper at temperatures between 70 and 180°C, preferably 130 – 140°C, using a nickel-copper matte. The copper sulphide fraction can be treated to recover copper by a leach-electrowin process and the nickel rich solution treated for the recovery of nickel metal by, for example, hydrogen reduction.
- Electrowin and electrostrip off the copper to concentrations below 0.3 g/l copper followed by, for example, cooling of the nickel rich electrolyte to precipitate nickel ammonium sulphate with the mother liquor returned to the leach of the residue in the initial precipitation step.
- Conditioning of the filtrate with, for example, chloride ions varying in concentration from 5 to 7 g/l, preferably 5 – 10 g/l for a period of time from ambient to 95°C, typically 90 – 95°C for 1½ hrs. The small quantities of PGM values in the leachate may be converted to chloro-complexes and become amenable to collection by ion exchange as described above. The base metal values remaining in solution may be recovered as described above.

The solids from the autoclave are dried to approximately 3-10% moisture using typical conventional dryers. The partially dried material is blended with concentrated sulphuric acid, typically in a cone mixer, which is heated indirectly to temperatures up to 350°C, preferably 280-310°C. The off-gas from this fusion step can be scrubbed to recover any products from the destruction of any CNO, CNS and CN compounds. The reaction temperatures can be maintained for periods of up to 1½ hrs.

The slurry from the fusion step is drained into a dilution vessel where it is diluted to typically 100g/l free sulphuric acid. The leach is maintained at temperatures up to 95°C for typically 1 hr and then filtered using any suitable solid-liquid separation. The solid

is washed before drying to produce a final concentrate rich in PGM values, typically assaying 15 – 85% PGM plus gold and silver, which may be refined in a conventional precious metal refinery to recover the PGM values.

The filtrate may be recycled to the repulping stage of the acidification residue.

In an alternative embodiment of the invention, pressure leaching of the acidification residue is conducted by first carrying out a caustic leach, followed by an acid leach (oxydrolisis), as shown in Figure 3. This alkali-acid combination leach is conducted to destroy the cyanide complexes and solubilise the base metals for subsequent recovery, typically as described above. The residue containing the PGM values may be reduction leached using a reductant such as formic acid to produce a high grade PGM concentrate suitable as a feed material to a conventional precious metal refinery.

A further alternative embodiment of the invention comprises a sulphuric acid fusion of the acidification residue followed by a chloride leach using reagents such as chlorine, hydrochloric acid and sodium chloride or a combination of some or all the reagents (Figure 3). The fusion/leach is conducted to produce a solution containing both the PGM values (as anions) and base metals (as cations). The PGM values are recovered by passing the solution through anionic ion exchange resin columns and followed by elution or alternatively ashing the resins. The base metal values are recovered by any suitable known method such as ion exchange, solvent extraction, precipitation or electrowinning.

In yet a further alternative embodiment of the invention, calcining of the acidification residue is conducted at a temperature between 250°C and 800°C, but preferably between 400-600°C, prior to an acid leach (Figure 3). The acid leach is conducted to solubilise the base metal values for subsequent recovery by any suitable known method. The PGM values remain in the residue and may be reductively leached to produce a high grade PGM concentrate.

Illustrative examples based on test work will now be presented to exemplify the present invention and should not be construed to limit the inventive method in any way. The

test work is presented below in a series of tests which have been conducted either using cyanide leachate, acidification solution or acidification residue.

Example 1: Precipitation with Sulphide at Raised Temperature

A test was conducted using NaHS to precipitate value metals from cyanide leachate at a raised temperature. 115% stoichiometric levels of NaHS were added and the temperature maintained at 90°C.

The test results are summarised in Table 1. It can be seen that good recoveries were achieved for Pt, Pd, Au, Cu and Co.

TABLE 1: Recoveries into Residue using NaHS

Metal	Pt	Pd	Au	Cu	Ni	Co
Recovery	97.5%	99.7%	99.5%	99.3%	11.8%	92.8%

Example 2: Precipitation with Sulphide at Ambient Temperature

A second series of tests was conducted on the cyanide leachate using NaHS over a range of pHs at ambient temperature to precipitate the value metals. 115% stoichiometric levels of NaHS were added and the reaction time was 5 minutes. Figure 4 shows that good recoveries were obtained for Cu, Co, Pd and Au at low pH values. The test results have been summarised in Table 2.

TABLE 2: Sulphide Precipitation Metal Recoveries

Precip pH	Cu (%)	Ni (%)	Co (%)	Fe (%)	Pt (%)	Pd (%)	Au (%)
0.5	99.77	11.98	95.45	0.00	47.65	99.46	98.10

1.5	99.97	31.02	92.39	71.42	38.44	31.55	86.52
3.5	99.75	2.45	4.03	46.77	1.20	0.86	15.87
5.5	95.66	0.04	1.76	14.15	0.35	19.86	20.42
7.5	0.02	0.00	0.00	0.00	0.00	28.29	23.06
9.5	0.00	0.00	0.00	0.00	0.45	26.36	39.41
11.5	0.00	0.00	0.75	0.00	8.03	15.35	6.87

Example 3: Precipitation using H_2O_2 and $Na_2S_2O_5$

A series of tests was conducted using H_2O_2 and $Na_2S_2O_5$ to precipitate the value metals from cyanide solution over a range of pH values. Tests were done using H_2O_2 only to precipitate the PGMs and base metals and these were followed with tests with sequential addition of H_2O_2 and $Na_2S_2O_5$ at ambient temperature at varying pH values.

Figures 5 and 6 show the effect of H_2O_2 only and a combination of H_2O_2 and $Na_2S_2O_5$, respectively, over a range of pHs on recoveries of PGMs and base metals. It can be seen that with the exception of Au, good recoveries of PGMs and base metals were obtained at pH values below 3.

Example 4: Acidification of Cyanide Leachate

In a series of tests, a cyanide leachate of a PGM containing concentrate was acidified with 50% H_2SO_4 over a range of pHs from 0 to 5. The temperature in each test was maintained at 45°C and the reaction time was 3 hours.

The concentration trends are shown in Table 3. PGM recovery to the precipitate was optimised at a pH of approximately 1.0. At pH2, the base metal residuals were at a maximum and then commenced redissolving. A similar trend was observed for the PGMs below a pH of 1.0. Figure 7 trends the soluble component at various pHs.

TABLE 3: Effect of Variable pH.

Test no	PH	Pt	Pd	Au	Cu	Ni	Co	Fe
		ppm						
Feed 1		4.21	5.58	1.95	1910	3970	17	32
dLeachate	4.95	4.56	5.85	0.98	1350	2875	26	27
Feed 2		3.42	2.62	0.75	862	1240	15	1250
Residue 1	3.15	3.04	2.31	0.62	87	102	32	2.8
Residue 2	2.41	2.66	2	0.67	1	1	6.8	2.3
Feed 3		3.3	3.53	1.3	1380	1950	50	6.8
Leachate 1	2.2	2.37	2.77	1.06	1.56	196	1.74	0.22
Leachate 2	1.95	0.64	1.35	1	3.53	132	0.55	0.17
Leachate 3	1.9	0.47	0.68	1	2.22	207	0.1	0.21
Leachate 4	1.42	0.26	0.37	0.61	1.51	343	0.15	0.29
Leachate 5	1.01	0.01	0.13	0.56	2.36	688	0.09	0.76
Leachate 6	0	0.41	0.25	0.57	9.76	872	0.23	0.57

Example 5: Recovery of PGM values using Ion Exchange Resin

In this test, filtered solutions from several individual small-scale pH2 acidification laboratory tests were initially passed through two columns in series containing 20 ml IRA 402 resin in each. The aim was to saturate and analyse the resin in the lead column.

Later a third column containing activated carbon and IRA AMBERLYST 40 (cation exchange resin) to extract gold was added.

The results from this test have been summarised in Table 4. It can be seen that an early break-through of gold from the first column occurred after only 15 200 ml had passed through the columns, and a break through of gold from the second column occurred after 37 500 ml solution through the columns.

Breakthrough of Pt and Pd occurred after some 74 280 ml solution passing through the columns and saturation of resin occurred at 133 130 ml.

The installation of the third column achieved its purpose of capturing the Au slippage from the IRA 402 resin columns.

The absorption of PGMs on the saturated resin when the first column was taken off line was 6,67 g/l Pt and 6,59 g/l Pd.

TABLE 4: Ion Exchange results from Acidification Filtrates

	Volume ml	bv Cum	Au	Pt	Pd	Cu	Ni	Co	
			mg/l						
Feed - 1			0.81	0.96	0.97	< 0.01	317	< 1.0	
Raffinate	0 - 5000	250	0.06	0.03	< 0.01	< 0.01	281	< 1.0	
Raffinate	8150 - 12050	603	0.03	0.01	< 0.01	< 0.01	291	< 1.0	
Raffinate	12050 - 15200	760	0.55	< 0.01	< 0.01	< 0.01	293	< 1.0	
Raffinate			0.03	<0.01	<0.01	<0.01	287	<1.0	lead
Raffinate	17500 – 19900	995	0.63	< 0.01	0.03	< 1	317	< 1.0	trail
Feed - 2			0.65	1.79	1.51	< 1.0	20	< 1	
Raffinate	35100 – 37400	1870	0.60	0.06	0.02	< 1	58	< 1	lead
			0.30	<0.01	0.02	<1	21	<1	trail
Feed - 3			0.78	0.16	0.71	< 1	86	< 1	
Raffinate	48530 – 51880	2594	0.62	0.01	0.01	< 1	60	< 1	
Feed - 4			0.73	0.54	1.05	2	41	< 1	
Raffinate	60880 – 62880	3144	0.98	0.01	0.03	< 1	314	< 1	
Feed - 5			0.93	0.94	1.06	< 1	393	< 1	
Raffinate	69280 – 74280	3714	1.07	0.32	0.15	< 1	433	< 1	
Feed - 6			0.65	0.55	0.88	< 1	341	< 1	
Raffinate	86240 – 87900	4395	1.62	0.47	0.81	< 1	307	< 1	
Feed - 7			0.91	0.28	0.22	< 1	300	< 1	
Raffinate	95380 – 98980	4949	0.87	0.59	0.74	<1	307	< 1	

Feed - 8			0.75	1.80	1.81	< 1	227	< 1
Raffinate	108230 – 111130	5557	0.93	0.41	0.8	< 1	228	< 1
Feed - 9			0.78	2.80	2.73	< 1	114	< 1
Raffinate	120920 – 123680	6184	1.2	0.65	1.19	< 1	7.7	< 1
Feed - 10			0.79	3.35	2.97	< 1	< 1	28
Raffinate	123680 – 127270	6364	0.95	0.72	0.65	< 1	< 1	< 1
Raffinate	127270 – 129960	6498	0.92	1.19	1.30	< 1	< 1	1.2
Raffinate	129960 – 131130	6557	0.91	1.66	2.32	< 1	< 1	20
Raffinate	131130 – 133130	6657	0.92	2.03	2.04	< 1	21	< 1
Raffinate			0.94	0.09	0.16	< 1	1.3	< 1
SATURATED RESIN A REMOVED FOR ANALYSIS - RESIN B MOVED TO A AND FRESH 20ml RESIN IN B								
Feed - 11			0.8	2.59	2.15	3.8	143	< 1
Raffinate	133130 – 137730	6887	< 0.05	0.24	< 0.05	< 1	148	< 1
INSERTED A THIRD COLUMN CONTAINING ACTIVATED CARBON AND IRA AMBERLYST 40 TO REMOVE GOLD SLIPPAGE								
Raffinate	0 – 4600	230	< 0.05	0.26	< 0.05	< 1	93	< 1
Raffinate	141780 – 145630	7282	0.78	0.09	0.11	< 1	151	< 1
Raffinate	8650 – 12500	625	< 0.01	< 0.01	< 0.01	< 1	148	< 1
Feed - 12			0.55	1.46	1.42	< 1	69	< 1
Raffinate	152550 – 155800	7790	0.71	0.07	0.13	< 1	72	< 1
Raffinate	19420 – 22670	1134	0.01	< 0.01	< 0.01	< 1	74	< 1
Feed - 13		1134						

lead
trail

Raffinate	161890 – 164910	8246	0.82	1.13	1.04	< 1	224	< 1
Raffinate	28760 – 31780	1589	0.06	< 0.01	< 0.01			
Feed – 14			0.59	4.50	2.55	1	141	1
Raffinate	173010 – 177060	8853	0.59	0.51	0.38	< 1	200	< 1
Raffinate	39880 – 43930	2197	0.10	< 0.01	< 0.01	< 1	191	< 1
Feed – 15			1.44	1.23	2.15			
Raffinate	177060 – 180780	9039	0.78	0.84	1.21	2.2	198	< 1
Raffinate	43930 – 47650	2383	0.17	< 0.01	0.01	< 1	180	< 1
Feed – 16			1.04	1.09	1.98	< 1	158	< 1
Raffinate	183120 – 187780	9389	0.66	0.60	0.45	< 1	210	< 1
Raffinate	49990 – 54650	2733	0.15	0.02	0.03	1.6	176	< 1
Feed - 17			0.65	1.01	1.83	< 1	238	< 1
Raffinate	190690 – 196405	9820	0.62	0.28	0.67	< 1	267	< 1
			0.47	<0.01	0.17	<1	246	<1
Raffinate	57590 – 63305	3165	0.24	0.02	0.27			

Note: analyses are for lead column if not specified

Example 6: Recovery of Residual PGMs from Leachate following an Acid Fusion

A series of tests were conducted to absorb PGM values onto an ion exchange resin from a solution following an acid fusion on acidification residue.

The residue was mixed with sulphuric acid, boiled down to white fumes for 30 minutes and diluted before chlorine was bubbled through at 70°C. A second set of tests was done by fuming with sulphuric acid followed by sodium chloride addition and boiling.

The resulting solutions from these tests were then passed progressively through a single column containing 10ml IRA 402 resin.

The results have been summarised in Table 5. It can be seen that selective extraction of PGMs took place over the base metals. However, Pt and Au absorbed preferentially to Pd once the resin became saturated. The analysis of the resin revealed values of Pt at 5,83 g/l and Pd at 6,68 g/l.

TABLE 5 : Ion Exchange results from Acid Fuming-Leach Filtrates

	Volume ml	bv Cum	Au	Pt	Pd	Cu	Ni	Co	Dissolution method
			mg/l						
Feed 1			0.17	12.9	25.1	8180	16400	457	H ₂ SO ₄ /Cl
Raffinate	0 - 80	8	< 0.01	1.83	0.59	7370	14680	417	
Feed 2			< 0.01	3.79	13.8	6420	8560	207	H ₂ SO ₄ /Cl
Raffinate	80 - 160	16	< 0.01	1.20	0.20	5910	9480	223	
Feed 3			1.10	10.1	22.9	5720	14300	232	H ₂ SO ₄ /Cl
Raffinate	160 - 235	24	< 0.01	0.91	0.20	5420	13400	223	
Feed 4			0.20	0.20	8.89	2545	5795	215	H ₂ SO ₄ /Cl ₂
Raffinate	235 - 310	31	< 0.01	0.36	0.15	2670	5850	201	
Feed 5			0.23	0.42	13.0	3990	3080	205	H ₂ SO ₄ /Cl ₂
Raffinate	310 - 385	39	< 0.01	0.23	0.14	3870	3430	205	
Feed 6			0.12	1.01	12.7	3060	2580	205	H ₂ SO ₄ /Cl ₂
Raffinate	385 - 460	46	< 0.01	0.21	0.15	3190	2650	206	
Feed 7			0.12	0.11	2.77	1210	2380	105	H ₂ SO ₄ /Cl ₂
Raffinate	460 - 535	54	< 0.01	0.08	0.07	1520	2440	117	
Feed 8			0.12	11.7	64.5	21058	23091	710	H ₂ SO ₄ /Cl ₂
Raffinate	535 - 660	66	< 0.01	0.31	1.32	19931	22021	673	
Raffinate	660 - 765	77	< 0.01	0.47	2.56	22545	25112	787	
Feed 9			1.53	39.0	64.5	12200	9600	3620	H ₂ SO ₄ /Cl ₂
Raffinate	765 - 845	85	< 0.01	0.28	0.64	13800	13800	410	
Feed 10			1.92	84.0	156	25300	38300	760	H ₂ SO ₄ /Cl ₂
Raffinate	845 - 985	99	< 0.01	0.23	1.51	24600	35000	720	

Feed 11			0.87	58.5	104	21900	22200	760	H ₂ SO ₄ /Cl ₂
Raffinate	985 - 1170	117	< 0.01	0.26	2.27	22100	23400	770	
Feed 12			1.29	32.0	46.0	11275	15274	432	H ₂ SO ₄ /Cl ₂
Raffinate	1170 - 1320	132	0.2	0.37	5.3	11197	15172	442	
Raffinate	1320 - 1540	154	0.08	0.31	9.7	11450	15395	435	H ₂ SO ₄ /Cl ₂
Feed 13			0.11	10.4	8.96	6360	7580	308	
Raffinate	1540 - 1740	174	0.05	0.10	13.3	6690	8320	325	H ₂ SO ₄ /NaCl
Raffinate	1740 - 1940	194	0.04	0.08	16.1	6380	7630	315	
Feed 14			0.75	30.5	25.0	10800	12000	610	H ₂ SO ₄ /NaCl
Raffinate	1940 - 2145	215	0.07	0.14	30.7	10800	12100	611	
Raffinate	2145 - 2315	232	0.02	0.36	43.7	11800	12400	638	H ₂ SO ₄ /NaCl
Feed 15			0.34	17.40	29.4	15000	15550	608	
Raffinate	2315 - 2515	252	< 0.01	1.22	51.3	14800	15100	633	H ₂ SO ₄ /NaCl
Feed 16			0.10	16.9	25.1	12000	13000	492	
Raffinate	2515 - 2745	275	< 0.01	0.78	31.5	12300	12300	497	

Example 7: Recovery of PGMs from High Pressure/Temperature Leach in a Two Stage Alkaline/Acid Medium followed by Ion Exchange.

A series of tests was conducted by subjecting the pH2 acidification residue to a two-stage alkaline/acid leach at varying pressures and temperatures from 10-20 bar (1000-2000 kPa) and 150-200°C, respectively.

The solutions generated by the pressure leaches were progressively passed through 10ml IRA 402 resin. The pH of the feed solutions varied between 0,49 and 5,83.

The results are summarised in Table 6. It can be seen that selective extraction of PGMs was achieved over the base metals. Pt and Pd extractions were good at all pH values, but Au slippage seemed more pronounced at high pH.

TABLE 6: Ion Exchange results from a two-stage Alkaline/Acid Leach filtrate

	Volume	bv	Leach		pH	Au	Pt	Pd	Cu	Ni	Co
			Press	Temp							
	ml	Cum	(bar)	°C	Feed	mg/l					
Feed 1			20	200	0.54	0.03	0.66	0.08	641	1065	16
Raffinate	2450 – 4600	460				< 0.01	< 0.01	< 0.01	634	1090	18
Feed 2			20	200	1.42	0.09	0.20	0.03	796	1180	42
Raffinate	6110 – 7290	729				< 0.01	< 0.01	0.01	832	1200	43
Feed 3			10	150	0.56	< 0.01	< 0.01	0.02	747	978	< 1
Raffinate	9460 – 10860	1086				< 0.01	< 0.01	0.02	777	1020	< 1
Feed 4			10	155	1.09	0.02	0.25	0.26	498	655	< 1
Raffinate	13020 – 13570	1357				< 0.01	< 0.01	0.05	501	700	< 1
Feed 5			10	155	1.28	0.31	0.43	0.80	1965	2060	< 1
Raffinate	15420 – 18030	1803				< 0.01	< 0.01	0.03	1720	1650	17
Feed 6			15	182	1.62	0.06	0.81	1.41	1700	1600	16
Raffinate	18030 – 21310	2131				< 0.01	< 0.01	0.02			
Feed 7			15	182	0.53	0.02	0.15	0.45	732	781	13
Raffinate	21310 – 24390	2439				< 0.01	< 0.01	0.02	850	847	13

Feed 8			20	200	0.49	0.29	0.06	0.12	2500	1810	80
Raffinate	24390 – 28150	2815				< 0.01	< 0.01	< 0.01	2460	1800	78
Feed 9			10	155	1.06	0.32	1.24	2.19	1700	3890	< 1
Raffinate	28150 – 30400	3040				0.04	< 0.01	< 0.01	1960	4350	3.3
Feed 10			10	155	0.94	0.29	0.26	2.07	1350	2030	2.7
Raffinate	30400 – 33800	3380				0.03	< 0.01	0.01	1790	2870	5.4
Feed 11			10	155	1.52	0.25	0.65	1.28	2660	3225	< 1
Raffinate	33800 – 35350	3535				0.03	< 0.01	< 0.01	2990	2690	< 1
Feed 12			20	200	0.66	0.07	0.09	0.06	1990	2340	83
Raffinate	35350 – 36650	3665				0.03	0.03	0.04	1990	2340	78
Feed 13			20	200	4.68	0.07	0.09	0.06	151	1800	68
Raffinate	36650 – 39560	3956				0.07	< 0.01	< 0.01	30	2580	80
Feed 14					1.02	0.11	0.65	1.58	8190	10500	222
Raffinate	39560 – 40810	4081				0.05	0.03	< 0.01	10200	14100	322
Feed 15			20	150	3.32	2.21	5.98	4.35	482	10100	15
Raffinate	40810 – 43660	4366				0.88	0.39	0.39	206	10120	< 1
Feed 16					0.72	1.17	1.53	0.51	7165	9935	19
Raffinate	43660 – 45210	4521				0.28	0.02	0.15	7180	9760	< 1
Feed 17					2.74	2.34	5.49	1.28	328	8480	13
Raffinate	45210 – 48810	4881				3.26	2.64	0.79	794	5290	13
Feed 18					5.83	1.88	3.64	1.59	515	18400	34
Raffinate	48810 – 50660	5066				2.80	2.32	1.06	335	18310	35

Feed 19					4.14	1.29	2.18	0.82	6870	20000	432
Raffinate	50660 – 52310	5231				0.40	0.06	0.04	6570	16800	513
Feed 20					1.92	1.47	1.04	0.23	13700	21000	524
Raffinate	52310 – 53620	5362				0.48	0.10	0.07	11500	15300	430
Feed 21					3.68	1.74	0.67	0.55	23700	28100	417
Raffinate	53620 – 56670	5667				0.40	0.08	0.09	18200	26650	463
Feed 22					0.69	0.82	0.62	0.42	22500	23700	350
Raffinate	56670 – 58350	5835						3	19400	24900	417
Feed 23					0.72	4.02	6.93	4.42			
Raffinate	58350 – 62100	6210				3.02	1.74	3			

Example 8: Recovery of PGM values from pH2 Acidification Residue by Calcining and Leaching

In this series of tests, a pH2 acidification residue was calcined at varying temperatures and then leached at high temperature and pressure. The calcine temperature varied from 250°C to 800° C and the calcined material was leached using dilute sulphuric acid at 6 bar (600 kPa) and 140°C for 4 hours.

Figure 8 shows the effect of calcining temperature on the extraction of value metals. It can be seen that good separation is effected at a calcine temperature between 400-600° C where the PGM values remain in the residue and the base metal values essentially report to the solution. The results are summarised in Table 7.

TABLE 7: Extraction into solution during Leaching after Calcining

Calcine.		Leach				Extraction into solution %						
Temp °C	Atm	H ₂ SO ₄ g/l	Pres kPa(t)	Temp °C	Time hr	Pt	Pd	Au	Cu	Ni	Fe	Co
250	Air	30.48	600	140	4	62.6	13.3	98.2	1	94.4	2.5	17.4
400	Air	30.48	600	140	4	7.1	2.7	8.4	99.5	99.6	19.8	98.3
600	Air	30.48	600	140	4	0.1	0.1	0.9	99.2	92.5	51.9	89.9
800	Air	30.48	600	140	4	1.1	0.4	10.1	97.0	88.5	45.1	75.6
800	CO	30.48	600	140	4	0.1	0.3	1.2	96.1	99.4	1.8	98.1

Example 9: Recovery of PGM values from pH2 Acidification Residue by Acid leaching and Fusion

In this series of tests, a pH2 acidification residue was leached with sulphuric acid under varying conditions of temperature and pressure over ranges of 165 – 185°C and 1200 – 1500 kPa(g), respectively, for 240 minutes. The residue from the leaching step was mixed with concentrated sulphuric acid and heated to 300°C for 60 minutes (fusion step).

The results are summarised in Tables 8 and 9. It can be seen from the tables that a 96% mass reduction was achieved at a temperature and pressure of 185°C and 1500 kPa(g). The concentrate produced had a PGM value of 29%.

TABLE 8: Leach and Fusion test conditions

Test	Leach				Fusion			Overall Mass Reduction
	Temp °C	Pres KPa(g)	Time min	Mass Reduction	Temp °C	Time min	Mass reduction	
1	180	1500	240	91.8%	300	60	80.9%	93.4%
2	180	1500	240	88.3%	300	60	74.4%	91.3%
3	165	1200	240	89.0%	300	60	69.1%	92.4%
4	185	1500	240	94.2%	300	60	67.1%	96.1%

TABLE 9: Analyses of products produced during Leaching and Fusion

Test	Stream	Pt (ppm)	Pd (ppm)	Au (ppm)
1	Leach feed	1100	1200	9.77
	Leach residue/Fusion feed	11500	12400	15.6
	Fusion residue	250000	199000	371
2	Leach feed	1100	1200	10.7
	Leach residue/Fusion feed	12300	12900	3.63
	Fusion residue	Insufficient sample		
3	Leach feed	1100	1090	9.66
	Leach residue/Fusion feed	10600	11800	5.27
	Fusion residue	61100	85200	109
4	Leach feed	1100	1090	9.66
	Leach residue/Fusion feed	20100	24500	11
	Fusion residue	121000	173000	165

DATED THIS 23rd DAY OF JULY 2003



SPOOR & FISHER
APPLICANT'S PATENT ATTORNEYS

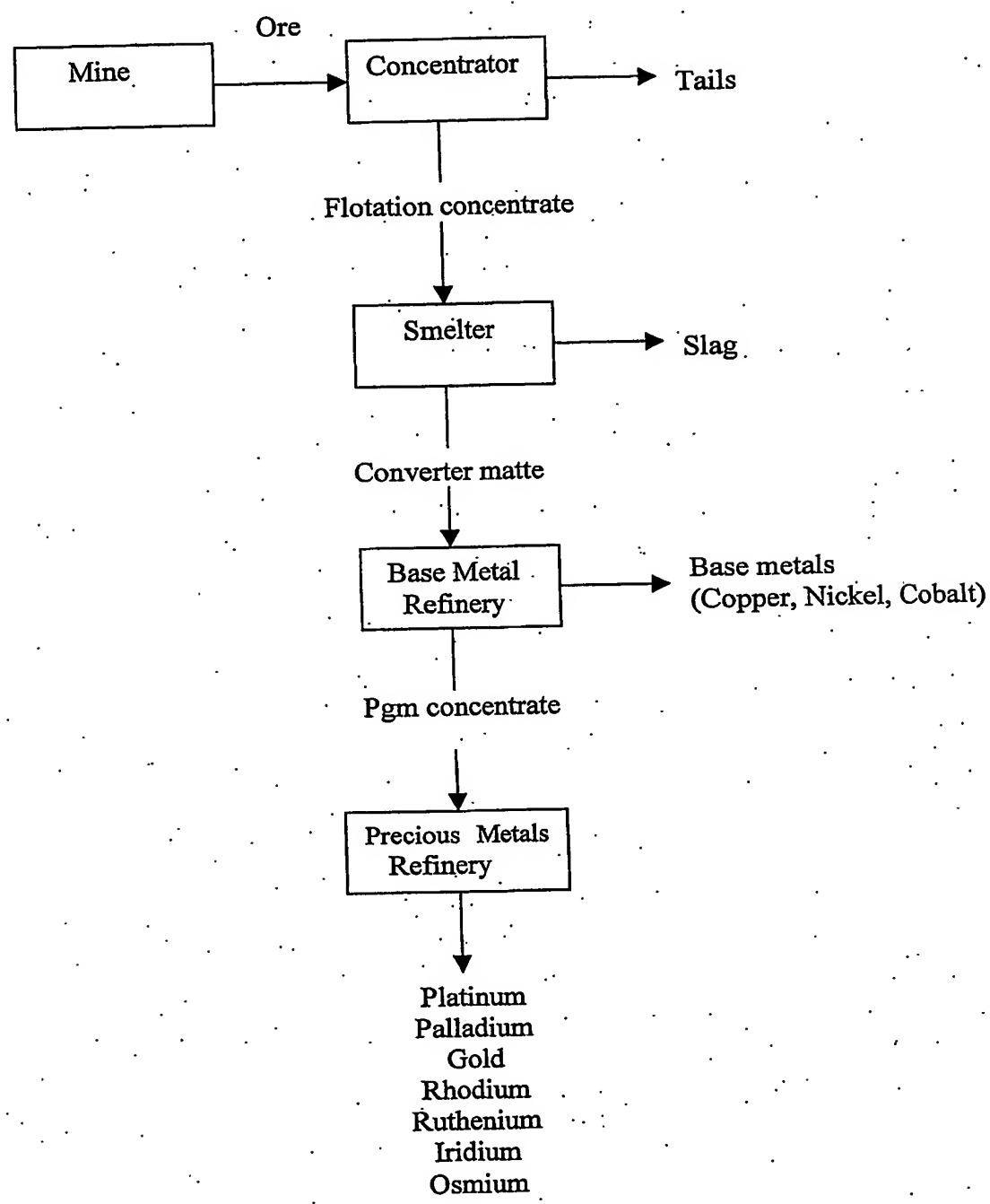


FIGURE 1

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6 SHEETS
SHEET 2

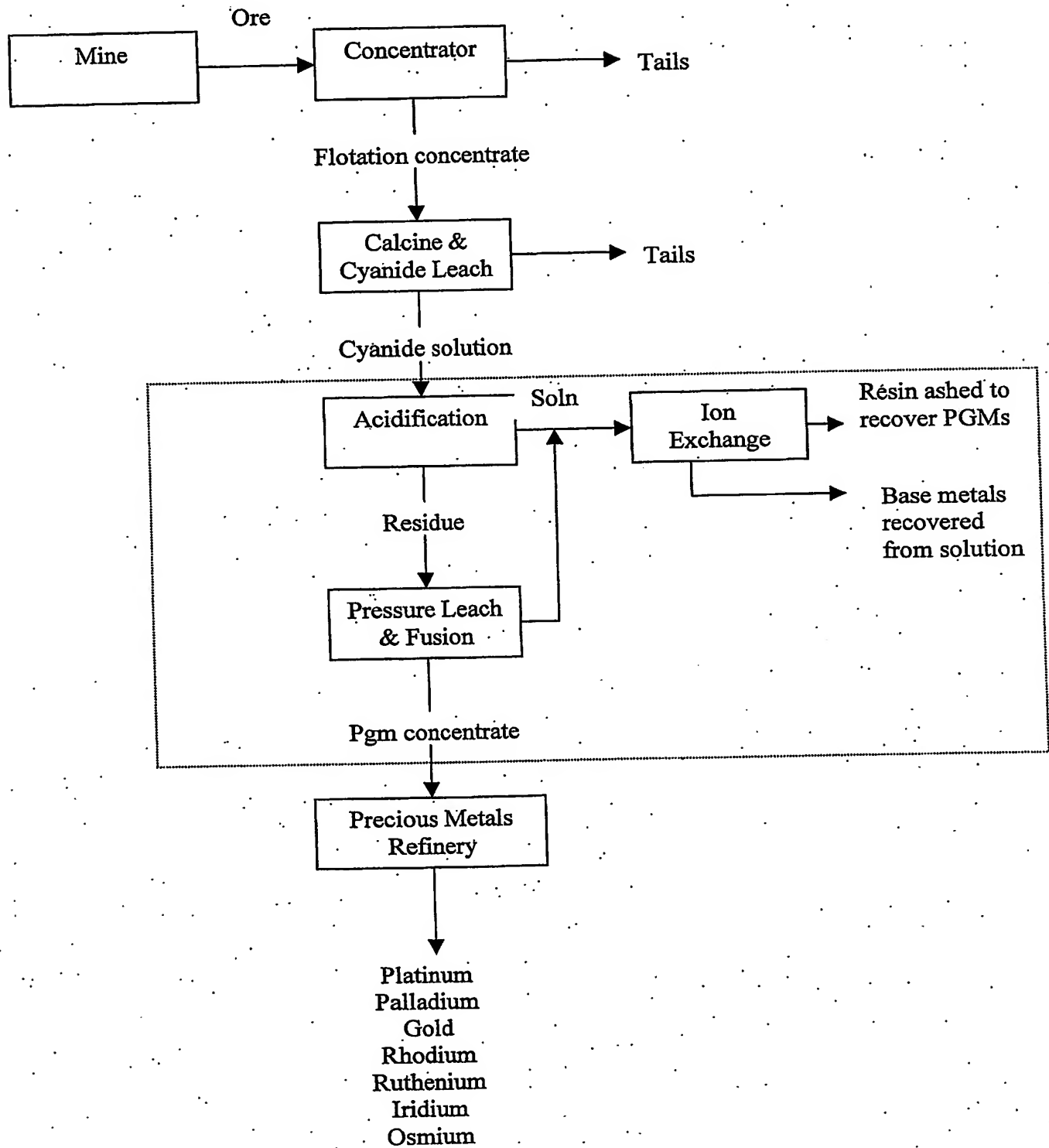


FIGURE 2

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6 SHEETS
SHEET 3

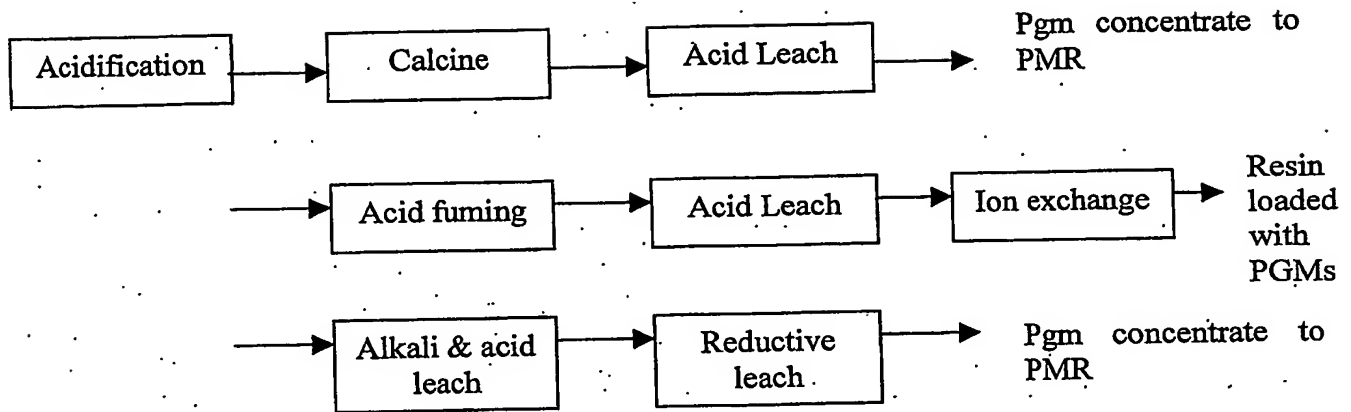


FIGURE 3

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6 SHEETS
SHEET 4

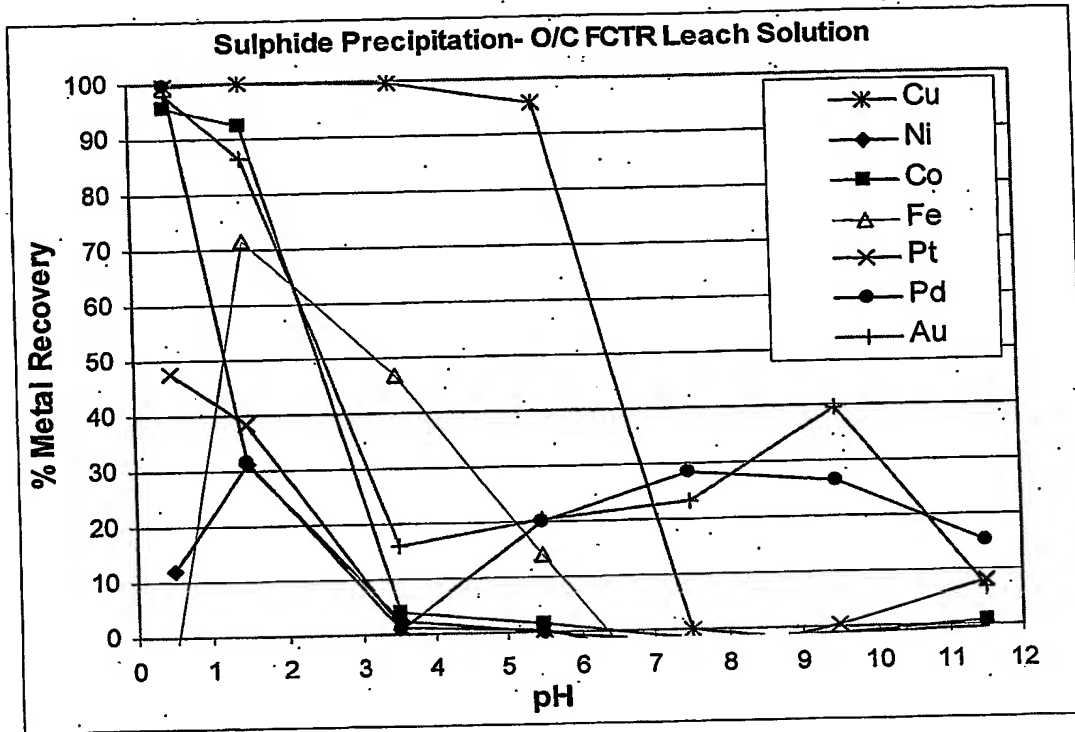


FIGURE 4

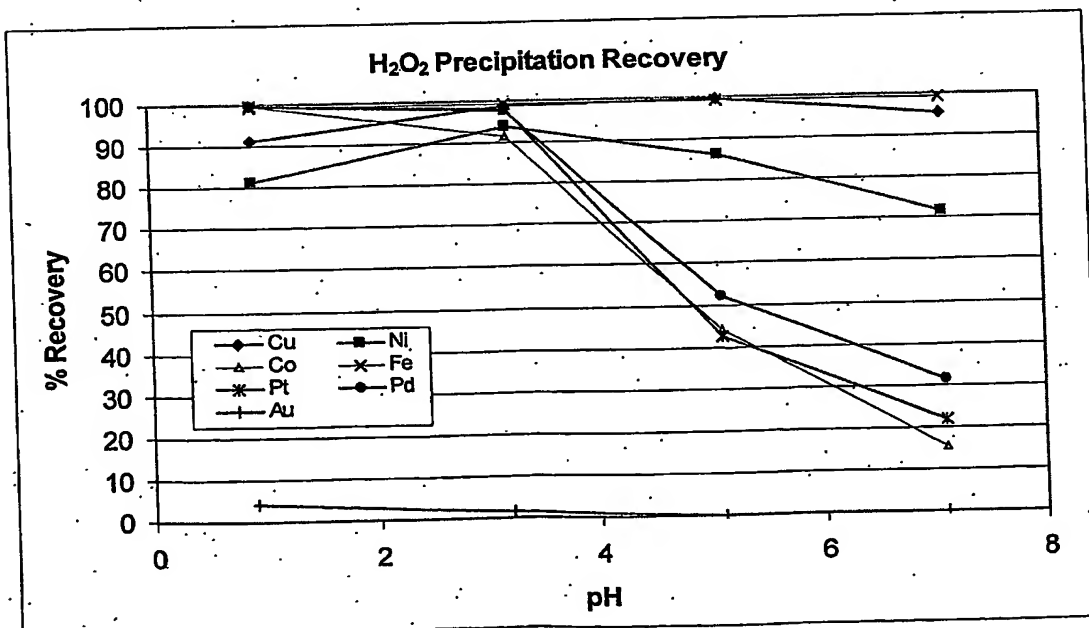


FIGURE 5

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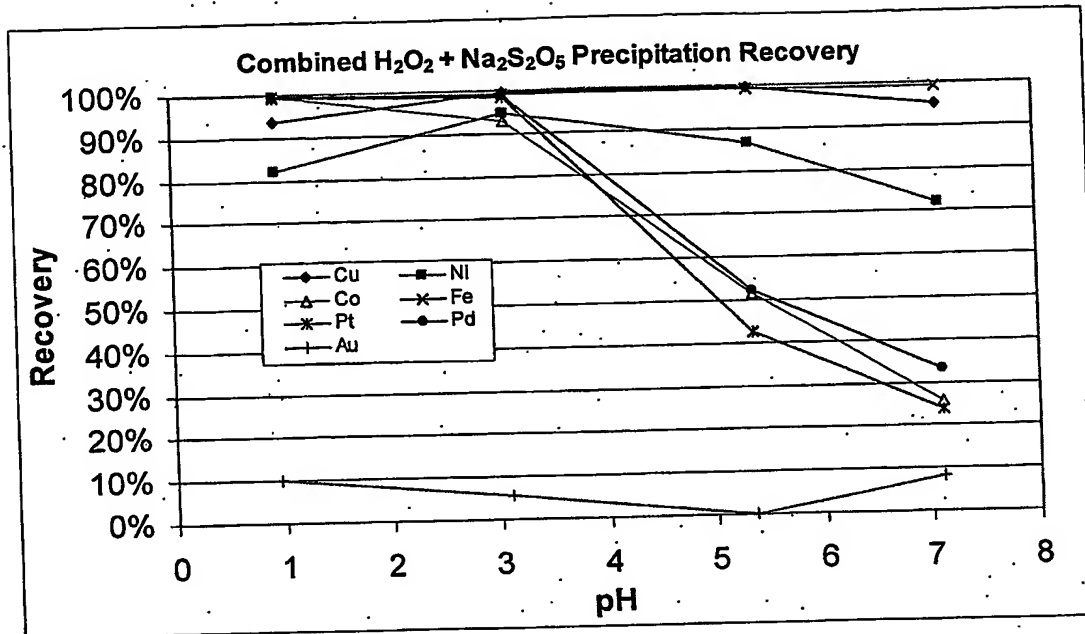


FIGURE 6

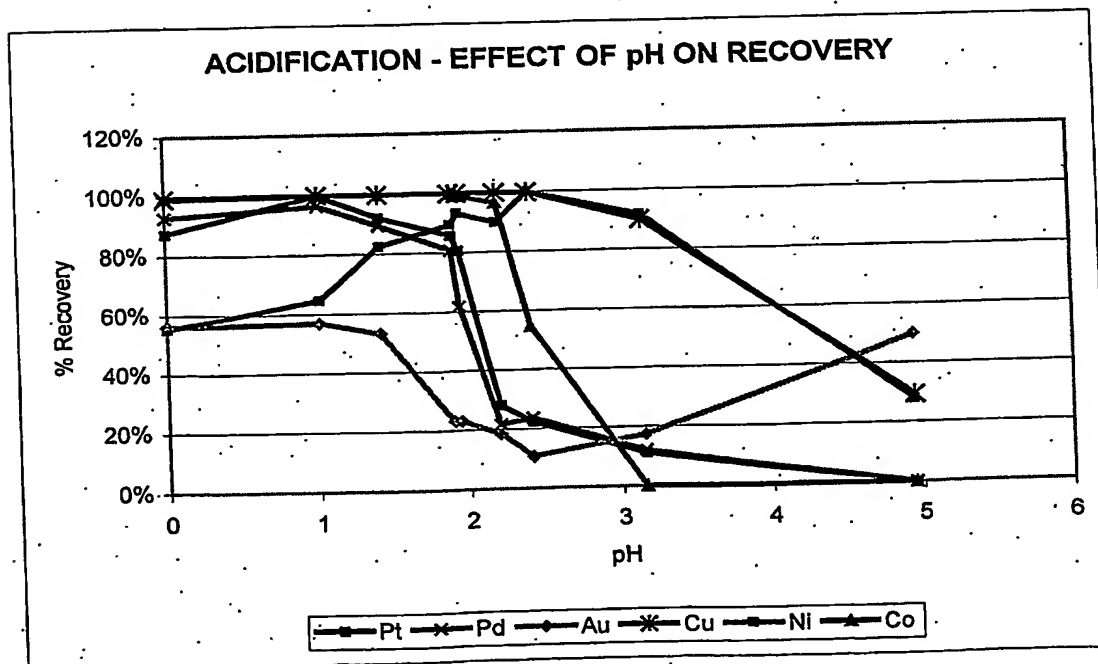


FIGURE 7

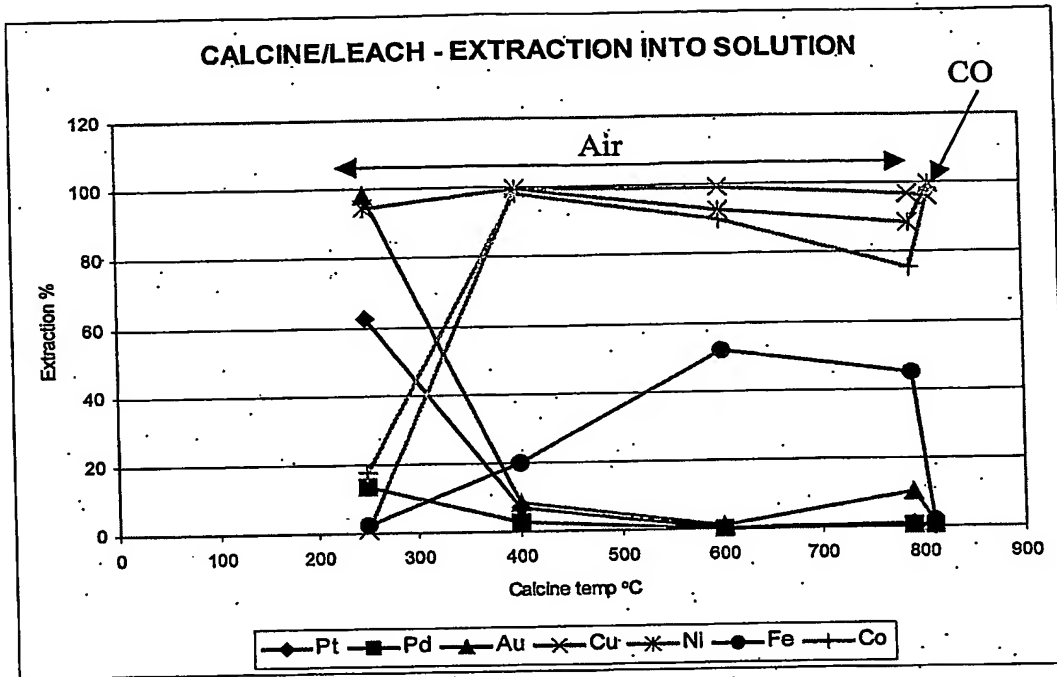


FIGURE 8

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/IB2004/001102

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C22B11/00 C22B3/20 C22B3/44

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C22B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, COMPENDEX, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	Claim 1; Column 3, line 4 - Column 4, line 11	8-15
Y	US 2002/152845 A1 (FLEMING CHRISTOPHER A ET AL) 24 October 2002 (2002-10-24) Abstract; Figure 1	8-10,12,13
Y	GB 1 153 717 A (TECHNION RES & DEV FOUNDATION) 29 May 1969 (1969-05-29) Claims 1 and 2	11,14,15
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☒ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

7 July 2004

Date of mailing of the international search report

20/07/2004

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INTERNATIONAL SEARCH REPORT

International Application No

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

International Application No

PCT/IB2004/001102

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